Oklahoma State University
Research Foundation

Semi-Annual Report #3
NASA Grant NsG-609

Project: Surface Parameters of Solids

Personnel:

Co-Investigators: Dr. E. E. Kohnke, Department of Physics
Dr. C. M. Cunningham, Department of Chemistry

Present Graduate Research Assistants:

H. Matthews - M.S. Candidate, Prospective Ph.D. Candidate, NASA Trainee

J. Rutledge, M.S. Candidate, Prospective Ph.D. Candidate, NASA Trainee

T. Vernardakis - M.S. Candidate

J. Tunheim - Prospective Ph.D. Candidate

Post-Doctoral Research Associate (June-August):

Dr. J. Houston

Degrees Granted:

J. Houston - Doctor of Philosophy (Physics), May, 1965.

H. Matthews - Master of Science (Physics), May, 1965.

The other members of the research group are making satisfactory progress toward the appropriate advanced degrees. Mr. J. Rutledge anticipates completion of all requirements for the degree of Master of Science by October, although it will not be officially awarded until the 1966 spring commencement.

Reports and Publications:

A Technical Report entitled, "A Preliminary Study of Certain Electrical Properties of Stannic Oxide Ceramics," by H. E. Matthews and E. E. Kohnke

(Interim Report SS-1) was furnished to the Office of Grants and Research Contracts in April, 1965. Word was received in June that it has been recommended for issuance as a formal NASA publication and has been turned over to NASA's Publication Standards Section for final preparation.

A paper entitled, "The Dispersion of Dielectric Constant and Resistivity in Stannic Oxide Ceramics," by H. E. Matthews was prepared and presented as a student exercise at the December, 1964 meeting of the Oklahoma Academy of Science. Proofs have been returned and the paper will appear in the next Annual Proceedings. Reprints will be furnished when they become available.

A Technical Report entitled, "Surface Parameters of Stannic Oxide in Powder, Ceramic, and Gel Form by Nitrogen Adsorption Techniques," is in the final stages of preparation and copies will be furnished to the Office of Grants and Research Contracts by the first of November.

Technical Accomplishments and Work in Progress:

A. Ceramic Specimen Preparation and Electrical Properties (MATTHEWS)

Preparation methods and preliminary electrical property evaluations were described in Interim Report SS-1 above. New methods for producing better samples are under continued study. A powder treatment using ethylene glycol as a binder solution has been developed. Very little densification was noted in the first samples composed of "pure" SnO_2, so grain sizes of the initial powder have been measured (~1.5μ) and an investigation of the possibility of using SnO_2 gel as a base material for higher density ceramics is under way. A limited study is being made on the kinetics of grain growth using existing data and metallograph pictures obtained at the Continental Oil Company Research Laboratory, Ponca City, Oklahoma.

Work is continuing on the properties described in the above report in an effort to correlate them with fabrication techniques. In particular, activation energies as obtained from conductance versus temperature curves in the temperature region from -150 to +100° C are being carefully investigated to determine the effects of ZnO doping.

B. Surface Areas (RUTLEDGE)

A conventional gas adsorption apparatus employing 32 volumes differing by about 8 cm^3 in the calibrated bulb system has been described in a previous status report and has been used to measure the volume of nitrogen adsorbed as a function of pressure on three forms of SnO_2 -- powder, ceramic and gel -- at 78° K. These isotherms were fitted to the B.E.T. adsorption equation with a good fit up to relative pressures of 0.20.

The specific surface of each sample was calculated from the B.E.T. equation and geometric considerations resulting in the following areas:

1) SnO_2 Powder (Reagent Grade) - 2.0 m^2/gm

2) SnO_2 Ceramic (Low Density) - 0.37 m^2/gm

3) SnO_2 Gel (Ion-free) - 173 m^2/gm.

In addition, the adsorption isotherms have been analyzed to determine the nature of the surfaces with respect to pores and capillaries. For the ceramic specimen, a Type II isotherm with no hysteresis indicates pore radii >300Å. The gel exhibited a Type I isotherm with no hysteresis suggesting small capillaries of molecular dimensions (i.e., ~ 20Å). The powder sample also exhibited a Type II isotherm indicating the average spherical diameter of the particles to be 0.43μ. Comparison with microscope data results in a roughness factor of about 3 for these particles.

This work forms the basis for Mr. Rutledge's M.S. thesis and will be reported in detail in the Technical Report mentioned previously.

C. Low Temperature IR Absorption (VERNARDAKIS)

An apparatus which can be used in the investigation of the photoconductivity of solids and the infrared absorption of gases adsorbed on these solids between 20 and 300° K has been assembled. This apparatus is now being checked out and it is first planned to investigate samples of SnO_2 gels which are being prepared in this laboratory.

Of initial interest will be the modification of the infrared transmission of the SnO_2 gel by the sorption of the hydrogen, oxygen, water vapor and other gases on its surface.

Developmental work is being done on the production of gels with different properties and a full report of preparation techniques will be included in the Technical Report referred to in the previous section.

D. Resonance Measurements (TUNHEIM)

Preliminary NMR studies have been made on water-saturated SnO_2 ceramics. A signal with large spinning side bands was observed for a white ceramic fired at 1100° C while no observable signal was obtained for a pink ceramic of the same composition fixed at 1460° C. Reasons for these results are not understood at this time and the phenomena are under continued investigation.

An NMR signal in SnO_2 gel dried at 120° C has been observed at room temperature with a $T_2 \tilde{}$ 5 m sec. Further studies varying drying conditions and total adsorption are in progress.

E. Photoresponse Studies

A portion of Dr. J. Houston's Research Associate stipend for the summer was supported by Grant funds. His efforts were directed toward extension and refinement of experimental techniques in the photoelectronic analysis work and toward improvements in circuitry and cryostat design. Added modifications of the basic cryostat design have largely eliminated electrical noise problems

and made possible an extension of the temperature range for these measurements. Completion of the new cryostat is expected shortly and a new series of photoelectronic analysis measurements will be initiated at that time.

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